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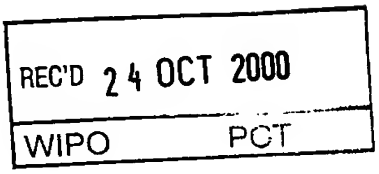

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NP10 8QQ

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I, the undersigned, being an officer duly authorised in accordance with Section 74(1) and (4) of the Deregulation & Contracting Out Act 1994, to sign and issue certificates on behalf of the Comptroller-General, hereby certify that annexed hereto is a true copy of the documents as originally filed in connection with the patent application identified therein.

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Signed P. Mahoney

Dated 24 July 2000

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Patents Act 1977
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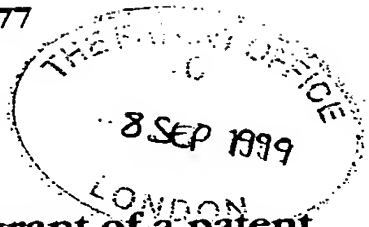

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1/77

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P01/7700 0.00 - 9921220.1

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

08 SEP 1999

The Patent Office

Cardiff Road
Newport
Gwent NP9 1RH

1. Your reference — PH99052

2. Patent application number *(The Patent Office will fill in this part)* — **9921220.1**

3. Full name, address and postcode of the or of each applicant *(underline all surnames)*

Rhone-Poulenc Agriculture Ltd
Fyfield Road
Ongar, Essex
CM5 0HW
ENGLAND

Patents ADP number *(if you know it)*

If the applicant is a corporate body, give the country/state of its incorporation — SGB3LN6001

4. Title of the invention — NEW HERBICIDAL COMPOSTIONS

5. Name of your agent *(if you have one)*

"Address for service" in the United Kingdom to which all correspondence should be sent *(including the postcode)*

Rachel M Colegate Hayward
Patent Department
Rhone-Poulenc Agriculture Ltd
Fyfield Road
Ongar, Essex
CM5 0HW

Patents ADP number *(if you know it)* — SGB2061001

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and *(if you know it)* the or each application number

Country	Priority application number *(if you know it)*	Date of filing *(day / month / year)*

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application	Date of filing *(day / month / year)*

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? *(Answer 'Yes' if:*

a) any applicant named in part 3 is not an inventor, or
b) there is an inventor who is not named as an applicant, or
c) any named applicant is a corporate body.
See note (d))

NO

PH99052

PATENTS ACT 1977

SPECIFICATION

BRITISH PATENT APPLICATION ENTITLED

NEW HERBICIDAL COMPOSITIONS

in the name of Rhône-Poulenc Agriculture Ltd, Fyfield Road, Ongar, Essex.

CM5 0HW. England

applying a herbicidally effective amount of the compounds, usually with an inert carrier or diluent, to the area where herbicidal control is desired. However, the herbicidal benzoylisoxazole and/or dione compounds have been found in some instances to adversely affect or interfere with the development of crop plants, especially maize crops. The effective use of these herbicides for controlling weeds in the presence of such crops may be enhanced by, or may require in certain instances, the addition of a compound which is antidotally effective with the herbicide.

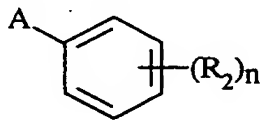
Although it is possible to say in general terms that herbicides may be used in the presence of a safener, the problem in identifying specific safeners for specific crops at appropriate rates to control weed growth, is substantial.

The applicants have found that certain compounds are effective antidotes for the protection of crops, especially maize crops, from herbicidal injury or the reduction of herbicidal injury caused by the application of an amount of a benzoylisoxazole and/or dione compound (optionally in admixture with a partner herbicide) effective to control the growth of weeds.

It is an object of the present invention to provide compositions of benzoylisoxazoles and/or dione herbicides in combination with antidotes, said compositions providing a reduction in crop injury, especially to maize (*Zea mays*), arising from the phytotoxicity of the herbicides.

Description of the Invention

The present invention provides a method of reducing phytotoxicity to a crop (especially maize) at a locus caused by the application thereto of a herbicidal benzoylisoxazole and/or dione derivative of formula (I):



(I)

in which the position of the carbonyl group and the group Q are reversed and the double bond in the ring is attached to the carbon atom attached to the group Q;

R represents a hydrogen atom or a halogen atom; a straight- or branched chain alkyl, alkenyl or alkynyl group containing from one to six carbon atoms which is optionally substituted by one or more halogen atoms; a cycloalkyl group containing from 3 to 6 carbon atoms optionally substituted by one or more groups R^5, one or more halogen atoms or a group $-CO_2R^3$; or a group selected from $-CO_2R^3$, $-COR^5$, cyano, nitro, $-CONR^3R^4$ and $-S(O)_kR^{13}$;

R^1 represents a straight- or branched-chain alkyl, alkenyl or alkynyl group containing up to six carbon atoms which is optionally substituted by one or more halogen atoms; or a cycloalkyl group containing from three to six carbon atoms optionally substituted by one or more groups R^5 or one or more halogen atoms;

R^2 represents a halogen atom; a straight- or branched-chain alkyl, alkenyl or alkynyl group containing up to six carbon atoms which is optionally substituted by one or more halogen atoms; a straight- or branched-chain alkyl group containing up to six carbon atoms which is substituted by one or more groups $-OR^5$; or a group selected from nitro, cyano, $-CO_2R^5$, $-S(O)_pR^6$, $-O(CH_2)_mOR^5$, $-COR^5$, $-NR^{11}R^{12}$, $-N(R^8)SO_2R^7$, $-N(R^8)CO_2R^7$, $-OR^5$, $-OSO_2R^7$, $-SO_2NR^3R^4$, $-CONR^3R^4$, $-CSNR^3R^4$, $-(CR^9R^{10})_t-S(O)_qR^7$ and $-SF_5$;

or two groups R^2, on adjacent carbon atoms of the phenyl ring may, together with the carbon atoms to which they are attached, form a 5 to 7 membered saturated or unsaturated heterocyclic ring containing up to three ring heteroatoms selected from nitrogen, oxygen and sulfur, which ring is optionally substituted by one or more groups selected from halogen, nitro, $-S(O)_pR^{13}$, C_{1-4} alkyl, C_{1-4} alkoxy, C_{1-4} haloalkyl,

or different groups selected from hydrogen, R^{23}, $-(CH_2)_uCO_2R^{22}$, halogen, cyano, C1-6 alkoxy, $-(CH_2)_x$-[phenyl optionally substituted by from one to five groups R^{24} which may be the same or different], and cycloalkyl containing from three to six carbon atoms optionally substituted by C1-6 alkyl or $-S(O)_pR^{21}$;

R^{20} represents phenyl optionally substituted by from one to five groups selected from halogen, C1-6 alkyl, C1-6 haloalkyl, C1-6 alkoxy and nitro;

R^{24} represents a group selected from halogen, R^{25}, nitro, cyano, $-CO_2R^{26}$, $-S(O)_pR^{21}$, $-OR^{21}$ and $-NR^{26}R^{27}$;

R^{25} represents a straight- or branched- chain alkyl group containing one to three carbon atoms optionally substituted by one or more halogen atoms;

R^{26} and R^{27} which may be the same or different, each represents hydrogen or R^{23};

p, q and u independently represent the values zero, one or two;

k and m represent one, two or three;

x represents zero or one;

t represents an integer from one to four; when t is greater than one, the groups R^9 and R^{10} may be the same or different;

or an agriculturally acceptable salt or metal complex thereof;

which method comprises applying to the locus of the crop an antidotally effective amount of an antidote compound, optionally with a partner herbicide.

It will be understood that the said antidote is, in general, antidotally effective for said benzoylisoxazole and/or dione derivative.

It will be understood that antidotes used in the method of the invention may form for example salts, and that the use of such salts is also embraced by the invention.

In this patent specification including the accompanying claims it is understood that the term 'agriculturally acceptable salts' is meant salts

Preferably in formulae (A-4) to (A-7), the groups R^{14}, R^{15}, R^{16}, R^{17}, R^{18}, R^{19} , R^{14a}, R^{15a}, R^{16a}, R^{17a}, R^{14b}, R^{15b}, R^{16b}, R^{17b}, R^{18b} and R^{19b} represent hydrogen or lower alkyl (preferably hydrogen, methyl or ethyl); L (in A-7a) represents NH; and Q represents hydroxy or -S-phenyl.

Compounds of formula (I) in which A represents (A-1),(A-2) or (A-3); R represents hydrogen or $-CO_2R^3$ (in A-1 or A-2) wherein R^3 represents or a straight- or branched chain alkyl group containing up to three carbon atoms; and R^1 represents cyclopropyl are preferred.

A further preferred class of compounds of formula (I) wherein A represents (A-1) are those wherein:

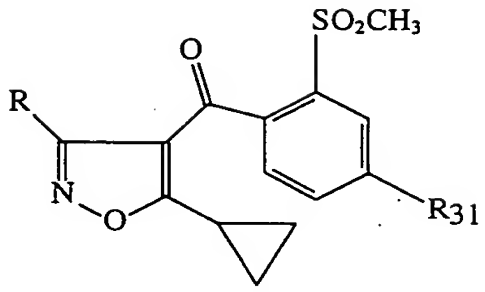
R is hydrogen or $-CO_2Et$;

R^1 is cyclopropyl;

and two groups R^2, on adjacent carbon atoms of the phenyl ring may, together with the carbon atoms to which they are attached, combine to form a 5 or 6 membered saturated or unsaturated heterocyclic ring which is fused to the 2,3 or 3,4 positions of the benzoyl ring; wherein the heterocyclic ring contains two hetero atoms selected from sulphur and oxygen which are attached to the 2 and 3, or 3 and 4 positions of the benzoyl ring; and in which the 4-substituent of the benzoyl ring is halogen or $S(O)_pMe$, or the 2-substituent of the benzoyl ring is methyl, $S(O)_pMe$ or $-CH_2S(O)_qMe$ respectively; and optionally the heterocyclic ring may be substituted by one or more halogen atoms.

A further preferred class of compounds of formula (I) are those wherein A represents (A-1); R is hydrogen or $-CO_2Et$; R^1 is cyclopropyl; R^2 is a halogen atom or a group selected from $-CF_3$, Me, Et, $-S(O)_pMe$, $-CH_2S(O)_qMe$ and optionally halogenated methoxy or ethoxy; and n is two or three.

A further preferred class of compounds of formula (I) wherein A represents (A-4) are those wherein:

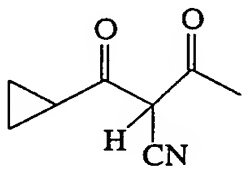


(Ib)

wherein R^{31} is chlorine, bromine or trifluoromethyl; and

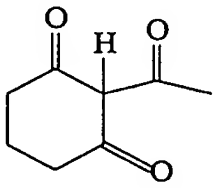
R is hydrogen or -CO_2Et.

Preferred diones are those in which a substituted phenyl ring as defined in formula (I); (Ia); or (Ib), is attached to a grouping;



Such diones in which the phenyl ring is substituted by two groups independently selected from halogen, alkyl, S(O)$_t$alkyl (t = 0, 1 or 2) or haloalkyl are also preferred.

Preferred triones are those in which a substituted phenyl ring, as defined above, is attached to a grouping;



The following compounds of formula (I) are among the most preferred for use in the present invention:

5-cyclopropyl-4-[2-chloro-3-ethoxy-4-(ethylsulphonyl)benzoyl]isoxazole;

4-(4-chloro-2-methylsulphonylbenzoyl)-5-cyclopropylisoxazole;

5-cyclopropyl-4-(2-methylsulphonyl-4-trifluoromethylbenzoyl)isoxazole;

4-(4-bromo-2-methylsulphonylbenzoyl)-5-cyclopropylisoxazole;

dichloroacetyl-3,4-dihydro-3-methyl-2*H*-1,4-benzoxazine; dichlormid, which is *N*,*N*-diallyl-2,2-dichloroacetamide; fenclorim, which is 4,6-dichloro-2-phenylpyrimidine; furilazole, which is (*RS*)-3-dichloroacetyl-5-(2-furyl)-2,2-dimethyloxazolidine; mefenpyr-diethyl, which is diethyl (*RS*)-1-(2,4-dichlorophenyl)-5-methyl-2-pyrazoline-3,5-dicarboxylate; CMPI, which is *N*-(4-chlorophenyl)maleimide; 4-hydroxy-1-methyl-3-(1-1*H*-tetrazol-5-ylmethanoyl)-1*H*-quinolin-2-one; daimuron, which is 1-(1-methyl-1-phenylethyl)-3-*p*-tolylurea; (S)-MBU, which is (S)-1-(1-alpha-methylbenzyl)-3-*p*-tolylurea; dimepiperate, which is S-1-methyl-1-phenylethyl piperidine-1-carbothioate; 5,5-diphenylisoxazolinone-3-carboxylic acid; and ethyl 5,5-diphenylisoxazolinone-3-carboxylate.

The mixtures of the invention may be used to obtain selective weed control with low crop injury in various crop plants such as maize, soybean, cotton, canola, sugar beet, potatoes, wheat, tobacco, rice and oil seed rape. Preferred crops include maize, sugar beet, cotton and canola. Particularly preferred crop species are maize and soybean, especially maize.

Effective weed control coupled with low crop injury is a result of treatment of a plant locus with a combination of a herbicidal benzoylisoxazole and/or dione derivative and an antidote compound in accordance with the present invention. By application to the 'plant locus' is meant application, for example to the plant growing medium, such as soil, as well as to the seeds, emerging seedlings, roots, stems, leaves or other plant parts.

The phrase 'combination of a herbicidal isoxazole and/or dione derivative and an antidote compound' includes various methods of treatment. For example, the soil of a plant locus may be treated with a "tank-mix" composition containing a mixture of the herbicide and the antidote which is "in combination" , or the soil may be treated with the herbicide and antidote compounds separately so that the "combination" may be made on, or in the soil. After such treatments of the soil with a mixture of herbicide and antidote or by separate or sequential application of the herbicide and the antidote to the soil, the herbicide

The antidote is applied in combination with the herbicide in a non-phytotoxic antidotally effective amount. By "non-phytotoxic" is meant an amount of the antidote which causes at most minor or no injury to the desired crop species. By "antidotally-effective" is meant an antidote used in an amount which is effective as an antidote with the herbicide to decrease the extent of injury caused by the herbicide to the desired crop species.

The following non-limiting examples illustrate the invention wherein Safener A is ethyl 5,5-diphenylisoxazolinone-3-carboxylate and Safener B is 5,5-diphenylisoxazolinone-3-carboxylic acid.

Example 1

Maize seeds were sown in non-sterile loam and safener, dissolved in acetone was applied to the soil surface. After 30 minutes a treatment of herbicide, Compound A [5-cyclopropyl-4-(2-methylsulphonyl-4-trifluoromethylbenzoyl) isoxazole] was applied to the treated soil.

A visual assessment of the percentage phytotoxicity (measured as a reduction in green plant matter or plant height) compared to an untreated control was made 14 days after treatment (DAT).

Maize seeds were sown in non-sterile loam and grown up to a 1.5 - 2 leaf stage. Safener, dissolved in acetone, was applied post-emergence to the soil surface. After 30 minutes a treatment of herbicide, Compound A [5-cyclopropyl-4-(2-methylsulphonyl-4-trifluoromethylbenzoyl) isoxazole] was applied to the treated soil.

A visual assessment of the percentage phytotoxicity compared with an untreated control was made 14 DAT.

in association with, and preferably homogeneously dispersed in, one or more compatible herbicidally- acceptable diluents or carriers and/or surface-active agents (i.e. diluents or carriers or surface-active agents of the type generally acceptable in the art as being suitable for use in herbicidal compositions and which are compatible with the herbicides of the present invention). The term "homogeneously dispersed" is used to include compositions in which the benzoylisoxazole and/or dione of formula (I) and antidote are dissolved in the other components. The term "herbicidal composition" is used in a broad sense to include not only compositions which are ready for use as herbicides but also concentrates which must be diluted before use.

The ratio of herbicide to antidote may vary depending upon the crop to be protected, weed to be inhibited, herbicide used, etc., but normally an herbicide-to-antidote ratio ranging from 1:25 to 60:1 parts by weight may be employed, although much higher rates of antidote may be used, e.g., 1:100 to 1:300 parts by weight of herbicide to-antidote. The preferred weight ratio of herbicide-to-antidote is from 1:10 to 30:1. Another preferred weight range ratio is from 1:1 to 20:1, with an even more preferred weight ratio range from 2:1 to 15:1.

Preferably, the compositions contain from 0.05 to 90% by weight of benzoylisoxazole and/or dione of formula (I) and antidote.

The herbicidal composition may contain solid and liquid carriers and surface-active agents (e.g. wetters, dispersants or emulsifiers alone or in combination). Surface-active agents that may be present in the herbicidal compositions of the present invention may be of the ionic or non-ionic types, for example sulphoricinoleates, quaternary ammonium derivatives, products based on condensates of ethylene oxide with nonyl- or octyl-phenols, or carboxylic acid esters of anhydrosorbitols which have been rendered soluble by etherification of the free hydroxy groups by condensation with ethylene oxide, alkali and alkaline earth metal salts of sulphuric acid esters and sulphonic acids such as dinonyl- and dioctyl-sodium sulphono-succinates and alkali and alkaline earth metal salts of high molecular weight sulphonic acid derivatives such as

additives such as antifoams, corrosion inhibitors, stabilisers, and water or an organic liquid in which the active substance is sparingly soluble or insoluble. Some organic solid substances or inorganic salts can be dissolved in order to assist in preventing sedimentation or as antifreeze for the water.

Application of the herbicide, antidote, or mixture thereof, can be carried out by conventional techniques utilizing, for example, hand-carried or tractor-mounted spreaders, power dusters, boom and hand sprayers, spray dusters, and granular applicators. If desired, application of the compositions of the invention to plants can be accomplished by incorporating the compositions in the soil or other media.

Herbicidal compositions according to the present invention may also comprise (a) and (b) in association with, and preferably homogeneously dispersed in, one or more other pesticidally active compounds and, if desired one or more compatible pesticidally acceptable diluents and carriers. Examples of other pesticidally active ingredients include fungicides, insecticides, plant growth regulators and, most preferably, herbicides.

The optional partner herbicides which may be combined with the derivatives of formula (I) and antidote are preferably selected from chloroacetamides (e.g. metolachlor, acetochlor, alachlor), sulfonylureas, thiocarbamates, dithiocarbamates, metribuzin, sulfentrazone, flumetsulam, clorasulam-methyl, oxasulfuron, flumiclorac, bentazon, chlorimuron, linuron, clomazone, dimethenamid, pendimethalin, trifluralin, clethodim and acifluorfen, bifenox, diflufenican, diuron, atrazine and ametryne.

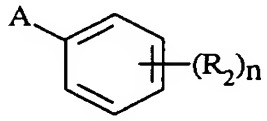
According to a further feature of the present invention there is provided a product comprising:

(a) a herbicidally effective amount of a benzoylisoxazole and/or dione derivative of formula (I),or an agriculturally acceptable salt or metal complex thereof; and

(b) an antidotally effective amount of an antidote compound or an agriculturally acceptable salt thereof;

CLAIMS

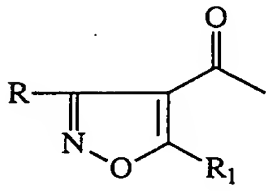
1. A method of reducing phytotoxicity to a crop (especially maize) at a locus caused by the application thereto of a herbicidal benzoylisoxazole and/or dione derivative of formula (I):



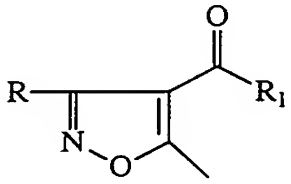
(I)

wherein:

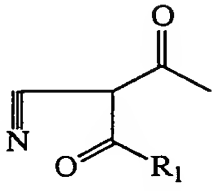
A represents a group (A-1) to (A-7):



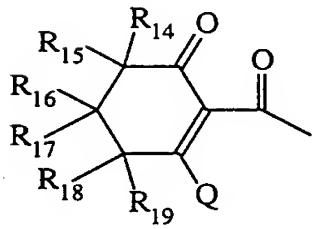
(A-1)



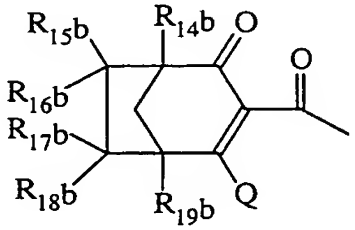
(A-2)



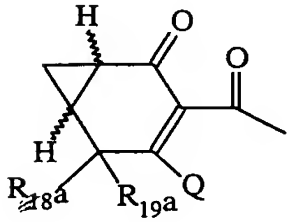
(A-3)



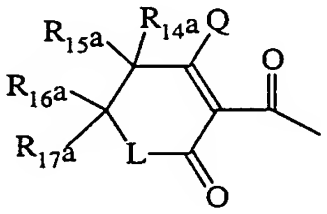
(A-4)



(A-5)



(A-6)



(A-7)

or a corresponding formula (A-6a) or (A-7a):

or two groups R^2, on adjacent carbon atoms of the phenyl ring may, together with the carbon atoms to which they are attached, form a 5 to 7 membered saturated or unsaturated heterocyclic ring containing up to three ring heteroatoms selected from nitrogen, oxygen and sulfur, which ring is optionally substituted by one or more groups selected from halogen, nitro, $-S(O)_pR^{13}$, C_{1-4} alkyl, C_{1-4} alkoxy, C_{1-4} haloalkyl, C_{1-4} haloalkoxy, =O (or a 5- or 6- membered cyclic acetal thereof), and $=NO-R^3$, it being understood that a sulphur atom, where present in the ring, may be in the form of a group -SO- or $-SO_2-$;

n represents an integer from one to five: when n is greater than one the groups R^2 may be the same or different;

R^3, R^4 and R^{22} each independently represent a hydrogen atom, or a straight- or branched chain alkyl group containing up to six carbon atoms which is optionally substituted by one or more halogen atoms;

R^5 and R^{23} each independently represent a straight- or branched-chain alkyl group containing up to six carbon atoms which is optionally substituted by one or more halogen atoms or a straight- or branched-chain alkenyl or alkynyl group containing from two to six (preferably from three to six) carbon atoms which is optionally substituted by one or more halogen atoms;

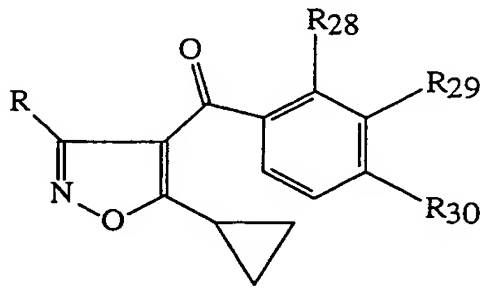
R^6 and R^7, which may be the same or different, each represent R^5; or phenyl optionally substituted by from one to five groups which may be the same or different selected from a halogen atom, a straight- or branched-chain alkyl group containing up to six carbon atoms which is optionally substituted by one or more halogen atoms, nitro, cyano, $-CO_2R^5$, $-S(O)_pR^{13}$, $-NR^{11}NR^{12}$, $-OR^5$ and $-CONR^3R^4$;

R^8, R^9 and R^{10} each represent a hydrogen atom or R^6;

R^{11} and R^{12} each represent hydrogen or R^5;

effective amount of an antidote compound, optionally with a partner herbicide.

2. A method according to claim 1 in which the isoxazole or dione herbicide has the general formula (Ia):



(Ia)

wherein:

R is hydrogen or -CO_2Et;

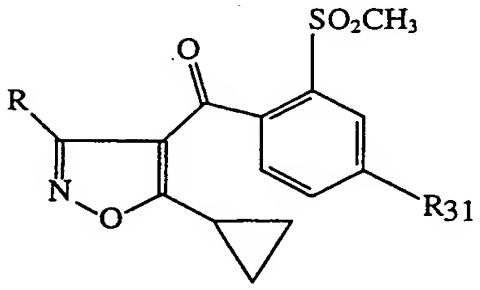
R^{28} is selected from -$S(O)_pMe$, Me, Et, a chlorine, bromine or fluorine atom, methoxy, ethoxy and -$CH_2S(O)_qMe$;

R^{29} is selected from a hydrogen atom, a chlorine, bromine or fluorine atom, methoxy, ethoxy and -$S(O)_pMe$;

R^{30} is selected from a hydrogen atom, a chlorine, bromine or fluorine atom, methoxy and trifluoromethyl; and

p and q independently represent the values zero, one or two.

3. A method according to claim 1 or 2 in which the isoxazole or dione herbicide has the general formula (Ib):



(Ib)

wherein R^{31} is chlorine, bromine or trifluoromethyl; and

R is hydrogen or -CO_2Et.

2-[2-nitro-(4-methylsulphonyl)benzoyl]-1,3-cyclohexanedione;

2-(2,3-dihydro-5,8-dimethyl-1,1-dioxospiro[4H-1-benzothiin-4,2'[1,3]dioxolan]-6-ylcarbonyl)cyclohexane-1,3-dione;

5-cyclopropyl-4-(2-methylsulphonyl-4-trifluoromethylbenzoyl)-3-methylthio-isoxazole;

2-cyano-3-cyclopropyl-1-(2-methylsulphonyl-4-trifluoromethylphenyl) propan-1,3-dione.

8. A method according to claim 17 in which the compound is 5-cyclopropyl-4-(2-methylsulphonyl-4-trifluoromethylbenzoyl)isoxazole or 2-[2-nitro-(4-methylsulphonyl)benzoyl]-1,3-cyclohexanedione.

9. A method according to claim 1 in which the antidote is selected from:

flurazole; fenchlorazole-ethyl; fenchorazole; benoxacor; dichlormid; fenclorim; furilazole; mefenpyr-diethyl; CMPI; 4-hydroxy-1-methyl-3-(1-1*H*-tetrazol-5-ylmethanoyl)-1*H*-quinolin-2-one; daimuron; (S)-MBU; dimepiperate; 5,5-diphenylisoxazolinone-3-carboxylic acid; and ethyl 5,5-diphenylisoxazolinone-3-carboxylate.

10. A method according to any one of the preceding claims in which the crop plant to be protected is maize.

11. A method according to claim 1 in which the application rate of the benzoylisoxazole and/or dione of formula (I) is from 0.004kg to 5kg per hectare.

12. A method according to claim 1 in which the application rate of the benzoylisoxazole and/or dione of formula (I) is from 0.01kg to 2kg per hectare.

13. A herbicidal composition comprising:

(a) a herbicidally effective amount of a benzoylisoxazole and/or dione derivative of formula (I) or an agriculturally acceptable salt or metal complex thereof, optionally in combination with a partner herbicide; and

(b) an antidotally effective amount of an antidote compound;

ABSTRACT

The present invention provides a method of reducing phytotoxicity to crops (especially maize) caused by a herbicidal benzoylisoxazole and/or dione derivative of formula (I) or an agriculturally acceptable salt or metal complex thereof; which method comprises applying to the locus of the crop an antidotally effective amount of an antidote compound, optionally with a partner herbicide.